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(12) UK Patent Application (19) GB (11) 2 027 627 A

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- (21) Application No 7925786  
(22) Date of filing 24 Jul 1979  
(23) Claims filed 24 Jul 1979  
(30) Priority data  
(31) 2833339  
(32) 29 Jul 1978  
(33) Fed. Rep. of Germany (DE)  
(43) Application published  
27 Feb 1980  
(51) INT CL<sup>3</sup>  
C21D 7/00 8/00  
(52) Domestic classification  
B3V 2C  
C7A 749 750 751 783  
78Y A249 A257 A25Y  
A28X A28Y A30Y A313  
A339 A349 A356 A35Y  
A37Y A38X A409 A439  
A459 A509 A51Y A525  
A53Y A541 A579 A587  
A58Y A609 A629 A671  
A673 A675 A677 A679  
A67X A681 A683 A685  
A687 A689 A68X A693  
A695 A697 A699 A69X  
A70X  
(56) Documents cited  
None  
(58) Field of search  
B3V  
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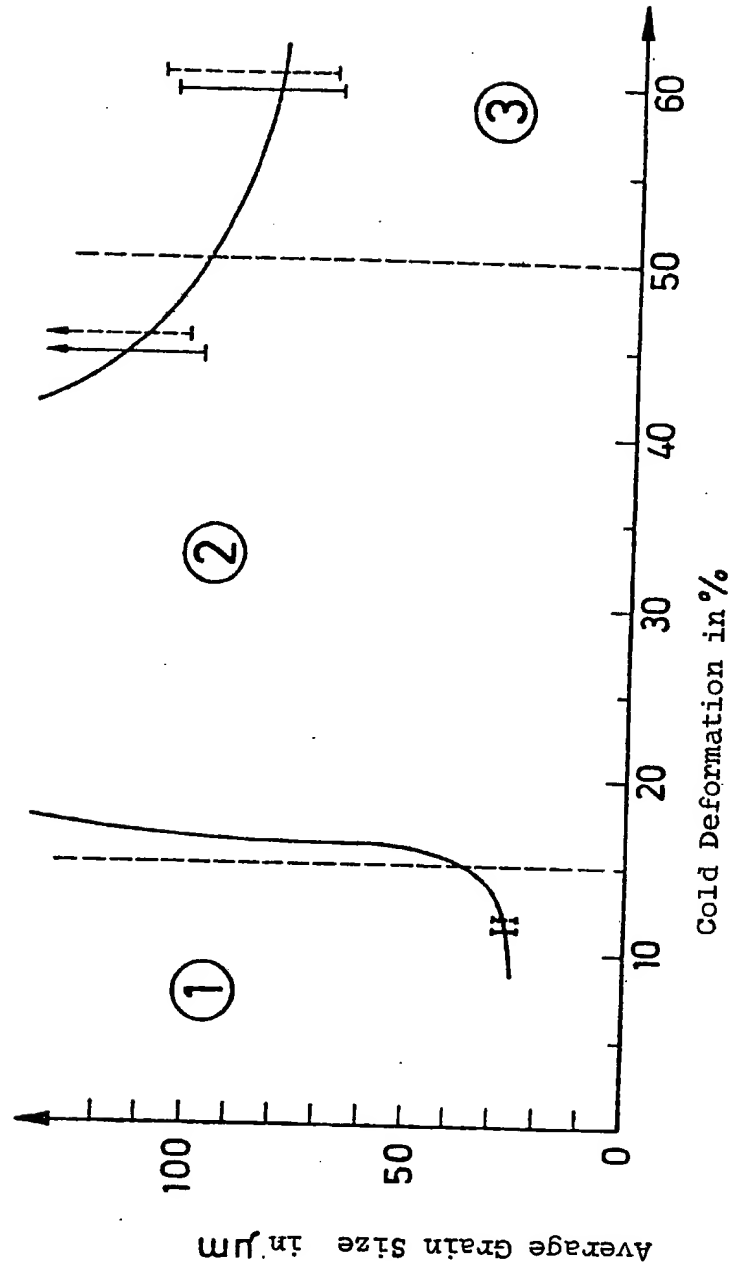
(54) Drawn pipes of austenitic  
chromium-nickel steels

(57) A method for improving the structure of such pipes made in a procedure which includes subjecting an unfinished pipe of such steel to an alternating succession of intermediate heat treatments and cold working, and then subjecting the pipe to a solution heat treatment, comprises carrying out the solution heat treatment at a

temperature of between 1353 and 1424°K and the final intermediate heat treatment at a temperature which is between 50 and 150°K lower than the temperature of the solution heat treatment and performing the final cold working after the final intermediate heat treatment, to produce a deformation having a maximum value of 15%, and as the last process step before the solution heat treatment.

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## SPECIFICATION

### Method for improving the structure of pipes drawn of austenitic chromium-nickel steels

#### BACKGROUND OF THE INVENTION

5 The present invention relates to a method for improving the structure of pipes drawn of austenitic chromium-nickel steels in which the pipes are subjected to a plurality of cold working and intermediate heat treatment steps as well as to a solution heat treatment. 5

In the production of pipes drawn of austenitic, titanium-stabilized steel, the unfinished pipes are subjected to a repeated alteration of cold working and intermediate heat treatment before the solution heat treatment is effected. In industrial manufacturing procedures, essentially a cold deformation of approximately 30% is effectuated during the last cold working step. The temperature for the intermediate heat treatments lies in the vicinity of the temperature for the solution heat treatment, for example, at 1353° K for the intermediate heat treatment and 1363° K for the solution heat treatment. 10 10

It has now been found that the properties of drawn pipes produced in this manner are insufficient for the particular use as cladding for nuclear fuels in a fast breeder reactor. Their creep resistance, ductility and resistance to fatigue under vibrational stresses are not high enough for the relatively long service life of the fuel elements. 15 15

#### SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to improve the properties of austenitic chromium-nickel steels during processing by increasing their creep resistance, ductility, resistance to fatigue under vibrational stresses, and/or their thermal stability even under unusual conditions, as for example under heavy neutron irradiation. 20 20

Briefly, these objects are achieved according to the invention by subjecting unfinished pipes drawn of austenitic chromium-nickel steel to a plurality of cold working and intermediate heat treatment steps as well as to a solution heat treatment. The final intermediate heat treatment step, preceding the solution heat treatment, is performed at a temperature that is between 50 and 150°K lower than the solution heat treatment temperature. The subsequent cold working step, which is the last process step before the solution heat treatment, is performed only to a maximum deformation of 15%. The solution heat treatment temperature is in the range from 1353 to 1423° K. 25 25

#### BRIEF DESCRIPTION OF THE DRAWING

30 The sole Figure shows an isothermal section of a large number of comparison test results for samples of pipes drawn of austenitic chromium-nickel steels according to the prior art and samples of pipes drawn of austenitic chromium-nickel steels produced according to the present invention. 30

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS

The method of the present invention is carried out in connection with a process in which an unfinished pipe drawn of austenitic chromium-nickel steel is subjected to an intermediate heat treatment at a temperature lying in the vicinity of the temperature for the final solution heat treatment, then in a second step the unfinished pipe is cold worked, and the intermediate heat treatment step and the cold working step are thereafter repeatedly alternated. Then, according to the invention, a last intermediate heat treatment step is carried out at a temperature that is between 50 and 150° K lower than the temperature for the final solution heat treatment. This is followed by a last cold working step whereby the unfinished pipe is cold deformed only to a maximum deformation of 15%. In the final step the pipe is subjected to a final solution heat treatment at a high temperature in the range from 1353 to 1423° K. 35 40

For a cold deformation of below 8%, the resulting structure is inhomogeneous. Between 15% and 50% cold deformation there occurs an undesirable secondary recrystallization. Above 50% cold deformation there begins the coarse grain range. For nuclear fuel cladding pipes a fine grain structure is desired because it alone imparts the optimum characteristics to the pipes during neutron irradiation. 45 45

The cold working steps are preferably in the range between 8 and 40%. The final cold working steps requires a cold working degree in the range between 8 and 15%. It is also possible to improve more the structure by a second final step. 50 50

In comparison tests, samples (A) were produced according to the prior art and samples (B) were produced according to the method of the present invention.

Both sample series were subjected to a final solution heat treatment at 1398° K and it was found that the samples (A) exhibited a secondary recrystallization in their structure involving grain sizes up to more than 0.5 mm, while samples (B) had a structure in the fine grain range with grain sizes of less than 40μ. Both sample series consisted of the austenitic steel 1.4970 (DIN x 10NiCrMoTiB1515) of a composition of 15 weight% Ni, 15 weight% Cr, 1.3 weight% Mo, 0.45 weight% Ti (=4 times the C content), 1.7 weight% Mn, 0.1 weight% C, 80 ppm B, and remainder iron. 55 55

Produce samples (A):

60 40 % cold working + intermediate heat treatment + 35 % cold working + intermediate heat treatment 60

+ 35 % cold working + intermediate heat treatment + final step 25 % cold working + solution annealing heat treatment.

Produce samples (B):

- 5 40 % cold working + intermediate heat treatment + 35 % cold working + intermediate heat treatment  
+ 35 % cold working + intermediate heat treatment + final steps 12 % cold working + intermediate heat  
treatment + 12 % cold working + solution annealing heat treatment. 5

The number of the intermediate heat treatments and cold working steps is without any influence to be improvement. The ranges of the permission degrees of deformation in the intermediate steps are preferably between 8 and 40 %. The first final step requires 8 to 15 %.

- 10 Several test series were performed utilizing solution heat treatment temperatures having various  
values between 1353 and 1398° K. The holding period for the solution heat treatment (time period  
after reaching the given solution heat treatment temperature) was varied between 15 minutes and two  
hours. The samples (B) were cold deformed by 12%. The last intermediate heat treatment temperature  
(T) and the holding period (t) before the last cold forming step was also varied as follows: 10

- 15 T: from 1253° K to 1373° K 15

t: from 15 minutes to two hours

The following table which shows a series of embodiments of the method according to the invention as well as the Figure will serve to explain the method of the invention in detail without being limited to the data given there.

- 20 In the table, the average grain sizes are given for the solution heat treated material which was  
subjected to a cold deformation of 12% before the solution heat treatment, these grain sizes being  
dependent on the intermediate heat treatment and solution heat treatment parameters. 20

TABLE  
Procedure sample A

Cold deformation [%]	Intermediate heat treatment parameters		Solution annealing heat treatment parameters		Average grain size [ $\mu$ m]
	T [K]	t [Min]	T [K]	t [Min.]	
12	1253	15	1353	15 ; 60 ; 120	22-27
		60		15 ; 60 ; 120	26-29
		120		15 ; 60 ; 120	28-31
12	1253	15	1398	15 ; 60 ; 120	27-32
		60		15 ; 60 ; 120	27-32
		120		15 ; 60 ; 120	31-33
12	1323	15	1373	15 ; 60 ; 120	28-31
		60		15 ; 60 ; 120	28-31
		120		15 ; 60 ; 120	28-31
12	1323	15	1398	15 ; 60 ; 120	27-37
		60		15 ; 60 ; 120	27-38
		120		15 ; 60 ; 120	27-38

Use for industrielle applications,

- 25 Time range for the intermediate heat treatment: 1253°K: 15—30 min 25  
1323°K: 5—30 min

Time range for the solution annealing heat treatment: 1373°K: 10—60 min  
1398°K: 5—40 min

TABLE  
Procedure sample B

Cold Working [%]	Intermediate heat treatment parameters		Solution annealing heat treatment parameters		Average grain size [ $\mu\text{m}$ ]
	T [K]	t [Min.]	T [K]	t [Min.]	
25	1253	15 60 120	1353	15 ; 60 ; 120 15 ; 60 ; 120 15 ; 60 ; 120	50 ; sr ; sr 50 ; sr ; sr 50 ; sr ; sr
25	1253	15 60 120	1398	15 ; 60 ; 120 15 ; 60 ; 120 15 ; 60 ; 120	sr ; sr ; sr sr ; sr ; sr sr ; sr ; sr
25	1323	15 60 120	1373	15 ; 60 ; 120 15 ; 60 ; 120 15 ; 60 ; 120	sr ; sr ; sr sr ; sr ; sr sr ; sr ; sr
25	1323	15 60 120	1398	15 ; 60 ; 120 15 ; 60 ; 120 15 ; 60 ; 120	sr ; sr ; sr sr ; sr ; sr sr ; sr ; sr

The Figure shows an isothermal section of a large number of test results and shows the recrystallization behavior (the respective grain sizes) of two heatings of the steel in question for the intermediate heat treatment parameters of 1253° K/60 minutes and the solution heat treatment parameters 1398° K/120 minutes in dependence on the degree of cold deformation in the last step before the solution heat treatment. Region 1 constitutes the range of stable and fine-grained structures; region 2 the range of occurrence of undesirable secondary recrystallization with grain sizes up to more than 0.5 mm; and region 3 the coarse grain range.

The solid lines 4, 6, 8 in the Figure shows the grain sizes for a second heat.

## 10 CLAIMS

1. In a method for improving the structure of drawn pipes made of austenitic chromium-nickel steels by subjecting an unfinished pipe of such steel to an alternating succession of intermediate heat treatments and cold working, and then subjecting the pipe to a solution heat treatment, the improvement wherein: said solution heat treatment is carried out at a temperature of between 1353 15 1423° K; the final intermediate heat treatment is carried out at a temperature which is between 50 and 150° K lower than the temperature of said solution heat treatment; and the final cold working is performed after the final intermediate heat treatment, is carried out to produce a deformation having a maximum value of 15%, and constitutes the last process step before said solution heat treatment.

2. A method for improving the structure of drawn pipes made of austenitic chromium-nickel steels, substantially as hereinbefore described.